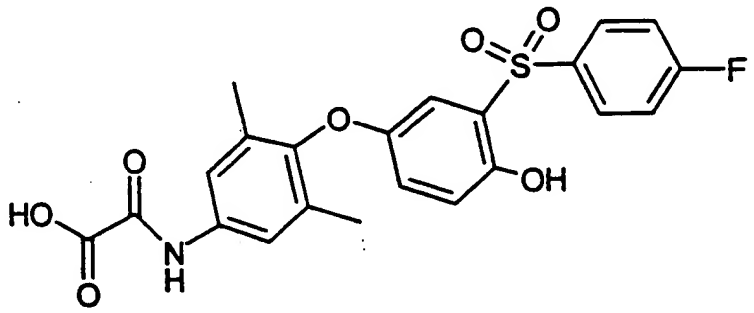
EXHIBIT

Substance Sheet

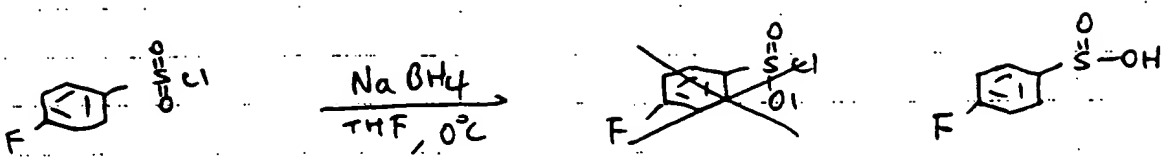
Labjournal ID:	U-0282-85-B	*Chemists:*	KUKKOLA, PAIVI WANG, HUA
Date:			
Department:	RES/MCD/MCDCHEM		
Chem. Code:			
US Sample No:			



MW Eff.:	459.45	*Subst./Stereo State:*	Single cmpd - achiral
Factor f:	1.000	*Struct. Assign.:*	Compatible with analytical data
MF Subst.:	$C_{22}H_{18}FNO_7S$		
MW Subst.:	459.45	*Approval Status:*	APPROVED
Initial Amount:	50 mg	*Synthesis Sheet:*	Yes
		End Product:	Yes
		Multi.Par./CChem.:	No
		To be sent to LFU/NCA:	No

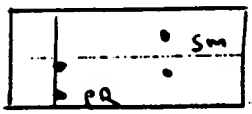
^{1}H-NMR:	Yes ; self service
MS:	2919-.
IR:	2919-.

Solvent System:	DMSO
Known in Lit.:	No;
To be tested on:	LPM; 4 mg, R. Steel
Clinical Codes:	
Comment Batch:	CHN; 100% purity by HPLC



	Amount	mmol	eq
4-fluorobenzenesulfonyl chloride (194.61)	500mg	2.57	1.0
Sodium borohydride (37.83)	0.49g	12.85	5.0
THF	20ml		

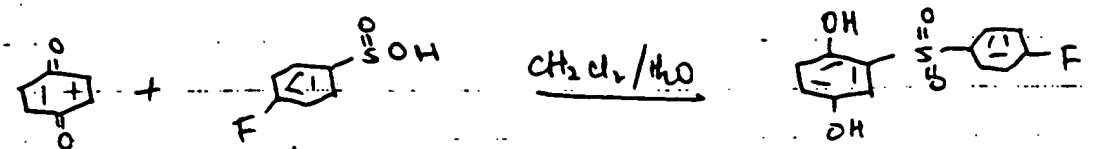
4-fluorobenzenesulfonyl chloride was dissolved in THF and NaBH4 was added in portions with stirring at 0°C. The rxn was stirred at 0°C for 1h, then RT for 5h.



hex/EtOAc 3:2

After removal of THF, H2O was added and mixture was acidified by the addition of HCl (6N) dropwise at 0°C. Extracted w/ EtOAc. Dried over Na2SO4 and concentrated to give UOSF2-67 as a white solid. 330mg

MS: $(M+)^-$ was detected



Ref. U0282-21

	Amount	mmol	g
U0282-67 (160)	320 mg	2.0	1.05
CH_2Cl_2	6 ml		
1.4-benzoquinone (108.10)	206 mg	1.9	1.0
H_2O	4 ml		

~~To a solution of U0282-67 in CH_2Cl_2, was added 1.4 benzoquinone~~ HJ

To a solution of 1.4-benzoquinone in CH_2Cl_2, was added a solution of U0282-67 in H_2O. The solution was stirred at RT. After 10 min, percipitation occured. The suspension was stirred at RT for 3 h.

It was filtered. The solid was collected. NB U0282-70 200mg as a off white solid

MS: ACN/H_2O/NH_4OH
$(2M-1)^-$ was detected

HJ

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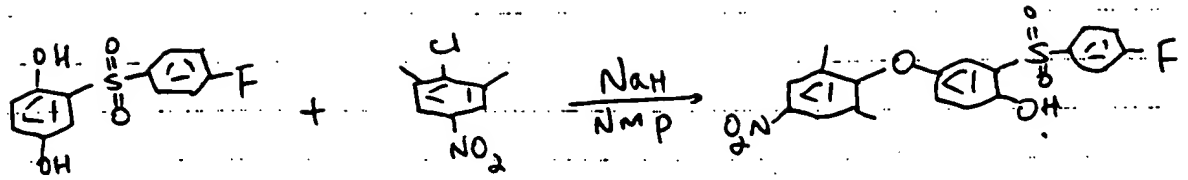
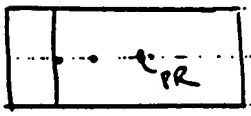


Ref U0282-73

	Ammount	mmol	eq
U0282-70 (268)	200 mg	0.75	1.0
NaH (24, 60%)	99 mg	2.46	3.3
NB 2694-12 (185.6)	167 mg	0.90	1.2
NMP	5 ml		

To a suspension of NaH in NMP at 0°C, was added U0282-70. The suspension was stired at RT for 30 min. NB2694-12 was added. The black suspension was stired at rt for 3h. It was quenched w/ H2O and extracted w/ Et2O (x3). The organic layer was washed w/ brine, dried over Na2SO4 and concentrated to give U0282-74 as a yellow solid.

MS: (M-1)⁻ was detected (416)



hex/EtOAc 3:2

The crude was purified w/ flash chromatography (hex/EtOAc 3:2) to give U0282-74A. 246 mg as a white solid.

¹HNMR (CDCl3) of U0282-74A: reasonablely clean

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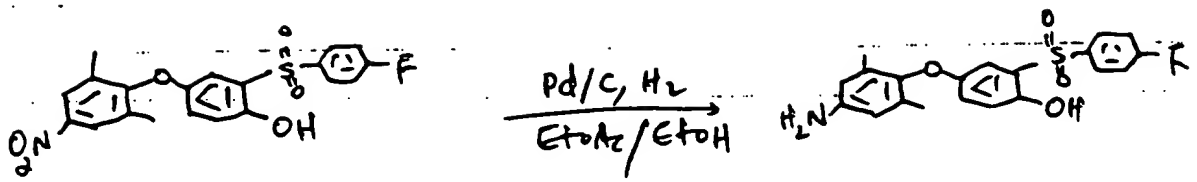
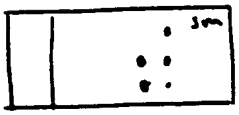


Ref. U0282-36

	Amount	mmol	eq
U0282-74A	240 mg		
Pd/C	24 mg		
EtOH/EtOAc	10 ml / 10 ml		

The rxn was stirred at RT & under H2 (1 atm) for 17 h.



hex/EtOAc 2:3

Small amount of starting material still present. It was stirred under H_2 balloon at RT for 4 h. It was completed by TLC. It was filtered through celite. The filtrate was concentrated to give U0282-80 190 mg as a solid.

^{1}HNMR (CDCl3): OK

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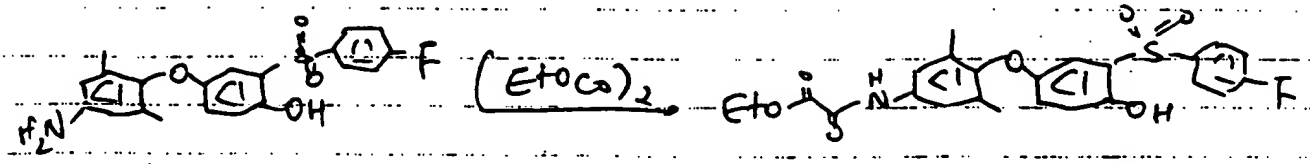

Ref. U0282-38

	Amount	mmol	eq
U0282-80	190mg		
$(EtOCO)_2$	2ml		

The rxn was stirred at 180°C for 3h. The solvents were remove by N_2 stream. Chromatographed on silica (hex/EtOAc 2:1→1) to give U0282-83A as a yellowish foam 195mg.

^{1}HNMR ($CDCl_3$): OK. trace amount of impurities.

Hua

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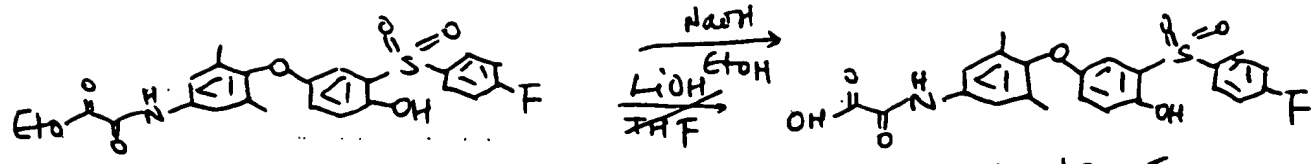
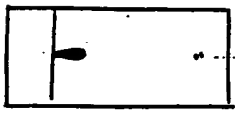


$C_{22}H_{18}NO_7SF$

Ref U0282-45

	Amount	mmol	eq
U0282-83 (487.5)	170 mg	0.35	1.0
NaOH (1M)	1.05 ml	1.05	3.0
EtOH	3 ml		

The rxn was stirred at RT for 2 h.



It was quenched w/ HCl (1N, 1.5ml). Extracted w/ EtOAc. The organ was washed w/ brine. Dried over Na_2SO_4 and concentrated to give U0282-85 as a foam.

U0282-85 was triturated w/ Et_2O/hexane. Dried to give U0282-85A 75 mg as a white solid.

^{1}HNMR of U0282-85A: some ether.

U0282-85A was dried in vacuum over at 50°C for 4 h. to give U0282-85 57mg as a white solid

^{1}HNMR ($CDCl_3$): OK.
MS: $(M-1)^-$ detected. CHN: OK.

50mg sample sent out.

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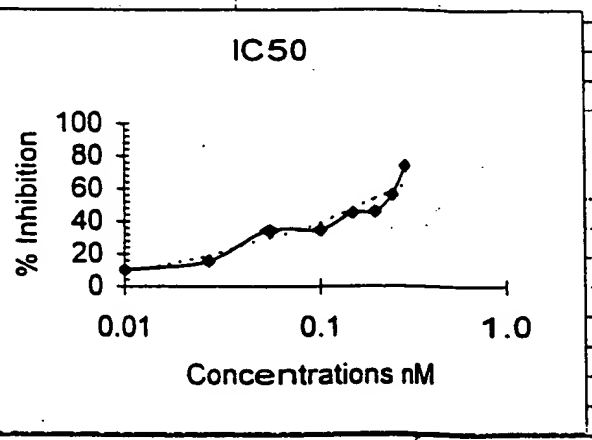
Sheet1

	nM	Reading1	Reading2	Mean	Activity(%)	Estimate	Hill Coefficient	IC-50
NO ENZ		494.00	494.00	494.00	0.00		0.84427425	0.167190443
NO INH		17268.00	17268.00	17268.00	100.00			
	0.010	15583.00	15583.00	15583.00	89.95	91.51		
	0.030	14660.00	14660.00	14660.00	84.45	81.01		
	0.060	11483.00	11483.00	11483.00	65.51	70.37		
	0.100	11450.00	11450.00	11450.00	65.32	60.68		
	0.150	9689.00	9689.00	9689.00	54.82	52.29		
	0.200	9572.00	9572.00	9572.00	54.12	46.23		
	0.250	7859.00	7859.00	7859.00	43.91	41.59		
	0.300	4881.00	4881.00	4881.00	26.15	37.90		

Concentrations	% Inhibition	Calculated
0.010	10.045	8.49
0.030	15.548	18.99
0.060	34.488	29.63
0.100	34.685	39.32
0.150	45.183	47.71
0.200	45.881	53.77
0.250	56.093	58.41
0.300	73.846	62.10



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Date